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13. ABSTRACT (Maximum 200 words)

The growth of thin films of single crystal h-GaN, h-AlN, and 3C-SiC on Si(100) and Si(111) with supersonic gas jets has been demonstrated. Among the major findings to date are the following. The growth rates on Si(100) are consistently 2-3 times higher than those on Si(111) for both GaN and AlN. Surface cleanliness affects significantly the growth rates. The quality of the films was uniformly better if the films were grown by atomic layer epitaxy than with concurrent dosing of the reactants. Kinetic energy of the reactants enhances significantly the growth rates. For kinetic energy higher than about 5 eV, however, the beginning of film degradation begins to appear from x-ray diffraction results. Single crystal h-GaN films have also been grown successfully on 3C-SiC initially deposited on Si(111), and similarly on h-AlN/Si(100). Large area growth of AlN on 4-inch diameter Si(100) wafers has been achieved using slit nozzle jets and a rotating substrate.



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The Growth and Characterization of GaN as a Photodetector

Objectives:

Optimization of GaN thin films grown by supersonic gas jet epitaxy on Si for photon capturing and emission devices is the primary goal of this project.

Progress:

A number of interesting results have been obtained in the growth of nitride thin films with supersonic jets. Our effort continues to focus on Si(111) and Si(100) as the substrate. Among the results we have obtained are: 1. ALE vs. concurrent dose; 2. differences in growth between Si(100) and Si(111); 2. importance of reactant translational energy; 3. changes in film quality as the thickness increases; and 4. preliminary results on film growth on 4-inch diameter Si(100) wafers.

Each film was characterized by x-ray diffraction with no monochromatization of the x-ray source or crystals for analyzing the diffracted beams. In addition, the film thickness was measured by the Alpha-Stepper and its morphology examined by an optical microscope. Real-time, in-situ optical reflectivity gave results on the film thickness in excellent agreement with those obtained by the Alpha-Stepper. A selected number of films were further characterized by SEM, atomic force microscopy (AFM), and Rutherford Backscattering Spectroscopy (RBS). The substrate temperature was 600-650 C during the growth; the temperature was determined by an infrared optical pyrometer through an infrared window. The chamber pressure rose from 2×10^{-9} Torr to 5×10^{-6} Torr during the growth for the growth of 1-cm diameter films and to 1×10^{-3} to 2×10^{-2} Torr for the growth of 4-inch diameter films. The growth rates lie in the range 0.05 to 0.15 $\mu\text{m}/\text{hr}$.

The growth of GaN from TEG and NH₃ leads to hexagonal (002) single crystal films only on Si(111) and only with ALE under the conditions we have attempted. In contrast, hexagonal single crystal AlN(002) films were grown only by ALE on Si(100) but both by ALE and concurrent doses on Si(111). The FWHM in the θ - 2θ scans of these films lie in the range 0.20-0.35 degrees, which were found to be the same as films grown by remote plasma MOCVD by NZ Applied Technologies and by low pressure MOCVD by APA Optics, Inc. on sapphire substrates. FWHM as small as 0.15 degrees has been achieved for AlN grown on Si(111). Under other conditions, polycrystalline films were implicated by x-ray diffraction. Furthermore, concurrent dose of TEG and NH₃ on Si(100) leads to columnar growth of GaN, with tall "spikes" of 40 μm in height above 0.5 μm thin film background; this columnar structure is suppressed with growth in the ALE mode. Such columnar growth is absent in AlN films. The growth rates on

Si(111) are consistently lower than those on Si(100) by a factor of about 2-3, indicating that the initial growth interface has an important effect on the subsequent growth.

Thin film membranes of GaN on Si were successfully fabricated. The diameters of these membranes approach those of the grown films (about 1 cm in diameter). The membranes were fabricated by etching away the silicon using HF:HNO₃:H₂O₂ solution which gave an extremely fast etch rate of Si without attacking the GaN. Etching of the AlN films was observed, however, in addition to Si; another solution should allow the fabrication of AlN membranes. The GaN membranes were extremely flat, indicating that the films are under tensile stress which is the desired stress for achieving flat membranes without any wrinkles in them.

The growth rate depends dramatically on the incident precursor translational energy. When the precursors were seeded in N₂, no measurable films were grown for both GaN and AlN. In contrast, under similar conditions, single crystal films were grown by seeding the precursors in He or H₂. The translational energies for NH₃, TEA, and TEG are 0.06 (0.21) eV, 0.37 (1.8) eV, and 0.43 (0.9) eV, respectively, when seeded in N₂ (He). Seeding in He versus H₂ results in very similar AlN films. The FWHM from θ-2θ scans, however, is larger for AlN films grown on Si(100) by seeding in H₂ compared to He (0.47° versus 0.32°). The translational energies for NH₃, TEA, and TEG seeded in H₂ are 0.37 eV, 4.9 eV, and 1.4 eV, respectively. The above values of translational energies are obtained for the specific growth conditions we have used for GaN and AlN film growths. The results indicate that the effects of translational energy are most important for energies between 0.5 eV and 1.0 eV for TEA and TEG and extra energies above this range do not affect the growth rate, and in fact may deteriorate the quality of the film as indicated by the FWHM of the θ-2θ scan. A critical threshold energy for achieving acceptable growth rate is about 1 eV.

The change in the quality of the films as a function of thickness was investigated for AlN on Si(100). For the 1 cm diameter films, "cracks" in the film are observed with optical microscopy for thickness greater than about 200 nm. The cracks are absent for film thickness less than 200 nm. The crack density increases as the thickness increases from 200 nm to 2.6 μm. For a 350 nm thick AlN(002) film on Si(100), the crack density is about 10⁶ cm⁻². As the film thickness increases, the θ-2θ scan changes from a single AlN(002) peak to a combination of AlN(002) and AlN(200) peaks and then to a single AlN(200) peak. The thickness at which the transition occurs is about 1 μm.

Single crystal 3C-SiC(111) thin films were grown successfully on Si(111) using dichlorosilane and acetylene in the ALE mode. The FWHM of the (111) diffraction peak in the θ-2θ scans in these films is about 0.5°. Hexagonal single crystal

thin films of GaN were grown successfully at NZ Applied Technologies by remote plasma CVD on the 3C-SiC/Si(111) grown at Cornell by supersonic gas jet epitaxy. This type of structure is ideal for making membranes of AlN since the SiC acts as an effective etch stop for HF:HNO₃:H₂O₂, the fastest etching solution for removing Si.

Initial success has been obtained in the growth of 4-inch diameter AlN films. The uniformity of the film, 450 nm thick, is excellent as indicated by RBS and there is not a single "crack" observed by optical microscopy over the entire Si(100) wafer. Two peaks were observed in the θ - 2θ scan, AlN(100) and AlN(110) for films grown by concurrent dosings of TEA and NH₃ on Si(100). Single crystal growth is expected in the ALE mode on Si(100). The ratio of Al:N as obtained by RBS is 1:1.3 compared to 1:1.1 for the 1 cm-diameter films. Variation of the growth parameters such as growth in the ALE mode and the growth temperatures, pressures, relative concentrations of TEA and NH₃ is expected to improve on the quality of these films.

Planned Work for Next Semiannual Period:

Thin membranes of GaN supported on Si will be fabricated to allow electrical and optical characterization of the films. Electrical leads will be connected to the GaN membranes to fabricate a simple large area photodetector. Subsequently, metallic patterns will be deposited on the membranes as a first step towards the fabrication of a two-dimensional image detector.

Alternative substrates will be explored, in particular, silicon-on-insulators, in order to improve the film properties. Ultrathin single crystal Si layer (< 200 Å) on SiO₂ will be fabricated starting with SOI, e.g. SIMOX wafers. The Si/SiO₂/Si-substrate structure will form a compliant substrate for the growth of GaN/SiC/Si/SiO₂/Si-substrate. The SiC will serve as a buffer layer between the nitrides and Si/SiO₂/Si-substrate.

An extensive ventilation system, sprinklers, and toxic gas detectors have recently been installed in our laboratory for the routine use of disilane (toxic) and flammable precursors (e.g., TEG, TEA). The laboratory is now upgraded and passes environmental and safety regulations.

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